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IS 6746 (1994): Unsaturated Polyester Resin Systems [PCD
12: Plastics]



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“Knowledge is such a treasure which cannot be stolen”

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भारतीय मानक

असंतप्त पालीस्टर रेजिन प्रणाली — विशिष्टि

(पहला पुनरीक्षण)

Indian Standard

**UNSATURATED POLYESTER RESIN
SYSTEMS — SPECIFICATION**

(First Revision)

UDC 678.674

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Plastics Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Unsaturated polyester resin systems are used predominantly in the construction of composites which have many and varied applications. Usually the resin systems are used with catalysts and ancillary chemicals to optimize the polymerization rate and processing characteristics. In some cases they may require a post-curing heat treatment. They are produced using a variety of starting materials to give a wide range of processing and end product properties, the latter also being significantly determined by the type and amount of reinforcement as well as the design of the component. Hence the properties determined by the introduction of any reinforcement using a particular manufacturing technique represent a result unique to that product.

While most of the polyester resins have quite a good shelf-life in storage, they will ultimately undergo changes to become unsuitable for usage over a period of time. The time for this change to occur depends on the nature of the resin and conditions under which it was stored. It is, therefore, important that the manufacturer's recommendations with regard to storage be observed to get maximum storage life. The curing agents (catalysts or initiator plus any accelerators or promoters) may also be affected by unsuitable storage, and it is important that these also be stored in accordance with the supplier's recommendations for the resin systems as a whole to perform at a reasonable degree of consistency within the storage life of the resin.

This standard was first published in 1972. In this revision classification of resin types used in the previous edition has been deleted which no longer can be exhaustive as a number of newer resin types with widely varying properties are continuously coming into the market. This allows the resins to be specified in accordance with the requirements set by the user for the envisaged application. Hence for the type of resin systems such as orthophthalic anhydride based, isophthalic acid based, bisphenol fumarate based, etc, the properties needed depending upon the requirements of the end applications has to be specified by the purchaser and the same has to be agreed between the purchaser and the supplier/manufacturer. In this revision four minimum properties have been identified and the limits of the nominal values prescribed, in addition to optional properties both for the liquid and polymerized (cured) resin systems. Allowable tolerances for various properties of liquid unsaturated polyester resins like colour, volatile content, viscosity, acid-value, gel time at 25°C and 82°C and for various properties of cured resins like Barcol Hardness, heat deflection temperature, water absorption and elongation at break have been prescribed. Requirements to be met for fire retardant grades to be classified as Type 1 or Type 2 have also been specified. Considerable assistance has been derived from BS 3532 : 1990 Method of specifying unsaturated polyester resin systems', issued by the British Standards Institution while preparing this revision.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

UNSATURATED POLYESTER RESIN SYSTEMS — SPECIFICATION

(*First Revision*)

1 SCOPE

This standard specifies the tolerances on the properties, classification of fire retardant grades and methods of sampling and tests for unsaturated polyester resin systems.

2 NORMATIVE REFERENCES

The following standards contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revisions, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

<i>IS No.</i>	<i>Title</i>
1070 : 1992	Water for general laboratory use (<i>third revision</i>)
2631 : 1976	Iso-propyl alcohol (<i>first revision</i>)
8543 (Part 4/ Sec 1) : 1984	Methods of testing plastics: Part 4 Short term mechanical properties, Section 1 Determination of tensile properties
13360 (Part 6/ Sec 6) : 1992	Plastics — Methods of testing : Part 6 Thermal properties, Section 6 Determination of flammability by oxygen index
13411 : 1992	Glass reinforced polyester dough moulding compounds

3 DEFINITIONS

For the purpose of this standard, the following definitions shall apply.

3.1 Resin System

The resin made by reacting saturated polyacids or anhydrides and unsaturated polyacids or anhydrides with polyols and dissolved in a suitable reactive monomer capable of undergoing crosslinking, and those additives that are added or supplied by the supplier to attain certain properties, constitute the resin system.

3.2 Batch

A quantity of material so designated by the supplier and substantially uniform.

3.3 Nominal Value

A typical physical property value or an average value of a range, indicated for a material by a manufacturer or supplier.

4 PROPERTIES

4.1 Minimum Requirements—Liquid Resins

The minimum requirements to be specified shall be viscosity, acid value, geltime at 25°C and volatile content. Nominal values for these properties shall be declared by the supplier. The limits on the nominal values shall be as given in Table 1.

4.2 Optional Properties—Liquid Resins

The properties mentioned in Table 2 are optional and are to be specified, if so desired by the purchaser. Nominal values for these properties shall be declared by the supplier. The limits on the nominal values shall be as given in Table 2.

NOTE —The purchaser should establish his own correlation between the properties of the processed articles and the material properties required for their achievement.

4.3 Optional Properties—Cured Resins

The measured values of the properties of the resins, as given in Table 3, cured as suggested by the supplier, shall not differ from the nominal values stated by the supplier by the limits given in Table 3.

Tests for these properties need not be carried out frequently, but, however, the supplier has to ensure that these properties conform to the limits given in Table 3.

4.4 Flammability of Cast Resins

Fire retardant resins have to meet both the requirements as given in Table 4, to be classified accordingly as Type 1 grade or Type 2 grade. This classification is based on both flammability tests and Critical Oxygen Index (COI) (though neither of them don't simulate actual fire conditions but to help in better screening of the materials).

4.5 Chemical Resistance

The chemical resistance, if desired by the purchaser, shall be subject to agreement between the

purchaser and the supplier and shall be determined by the method given in Annex P.

4.6 Shelf Life of the Resin System

When stored in the original unopened containers under the conditions specified by the manufacturer, the material shall comply with the requirements of this specification, with the exception that the change in geltime and viscosity shall not be greater than 30 percent of initial measured value, throughout the period stated by the manufacturer which shall not be less than 3 months.

NOTE — The criteria for assessing when the resin becomes unsuitable are given to assist the user to determine this point. The resin may even then still be used with reference to the manufacturer or supplier, since under favourable storage conditions the useful life of the resin may significantly exceed the three months specified.

5 PACKING AND MARKING

5.1 The material shall be supplied in packages as agreed to between the purchaser and the supplier. The following information shall be appropriately indicated on the packing:

- a) Name and grade reference;
- b) Batch number;
- c) Indication of the source of manufacture or trade-mark, if any; and
- d) Date of manufacture and guaranteed shelf-life.

5.2 The package may also be marked with the Standard Mark.

6 INSTRUCTIONS FOR USE

Manufacturer shall provide the following information to the purchaser:

- a) *Method of Use*—Addition levels of catalysts, accelerators or promoters to the resin and

the working temperature required to achieve optimum curing.

- b) *Precautions* — Details of the safety precautions to be taken during storing, handling, mixing and curing the resin and curing agents.
- c) *Storage Conditions* — Details of recommended storage conditions.

7 SAMPLING

7.1 Owing to the possibility of stratification within the resin in the container it is essential that the contents of any container selected for sampling for test purpose shall be mixed thoroughly before the sample is taken.

7.2 Criteria for Conformity

These resin systems shall be deemed to comply with this standard if they comply with all the requirements of Table 1 and Table 4 in addition, in the case of fire retardant grade resins.

8 METHODS OF TEST

8.1 Tests shall be conducted as prescribed in Annexes A to P.

8.2 Preparation of Test Specimen

Resins shall be cast in accordance with Annex P and the test pieces shall either be cut or machined from the cast sheet as per the required dimensions of the test methods.

8.3 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1992) shall be employed.

NOTE — ‘Pure chemicals’ shall mean chemicals that do not contain impurities which affect the results of analysis.

Table 1 Limits for the Minimum Property Requirements for Unsaturated Polyester Resin Systems (Clauses 4.1 and 7.2)

Sl No.	Property	Units	Limits	Test Method
(1)	(2)	(3)	(4)	(5)
i)	Viscosity at 25°C			
	a) Thixotropic (Low shear and High shear)	Centipoise	± 30%	Annex A
	b) Non-thixotropic	do	± 20%	do
ii)	Acid value	mg KOH/g	± 4	Annex B
iii)	Volatile content	%	± 3	Annex C
iv)	Geltime at 25°C	Min	± 20%	Annex D

Table 2 Limits for the Optional Properties of Liquid Unsaturated Polyester Resin Systems
(Clause 4.2)

Sl No. (1)	Property (2)	Units (3)	Limits (4)	Test Method (5)
i)	Colour	a) Hazen b) Gardner	State maximum value do	Annex E
ii)	Relative density at 27/27°C	—	± 0.01	Annex F
iii)	Refractive index at 25°C	—	± 0.000 2	Annex G
iv)	Geltime at 82°C	Min	± 20%	Annex H
v)	Hydroxyl value	mg KOH/g	± 3	Annex L
vi)	Styrene emission	g/m ² at 60 minutes	State maximum value	Annex M

Table 3 Limits for the Optional Properties of Cured Unsaturated Polyester Resin Systems
(Clause 4.3)

Sl No. (1)	Property (2)	Units (3)	Limits (4)	Test Method (5)
i)	Barcol hardness	BHU	± 5	Annex J
ii)	Heat deflection temperature	°C	-2 +10	Annex H of IS 13411 : 1992
iii)	Water absorption after 7 days at 25°C	mg %	±10 or ± 0.1	Annex K
iv)	Tensile elongation at break	%	- 0% + 25%	IS 8543 (Part 4/ Sec 1) : 1984 (using Type 1 test specimen)

Table 4 Categorization of Fire Retardant Resin Grades
(Clauses 4.4 and 7.2)

Sl No. (1)	Property (2)	Type I II		Test Method (5)
i)	Flammability	Very low	Low	Annex N
ii)	Critical Oxygen Index (COI)	28 Min	24 Min	IS 13360 (Part 6/ Sec 6) : 1992

ANNEX A

[Table 1, Sl No. (i)]

MEASUREMENT OF VISCOSITY BY BROOKFIELD VISCOMETER

A-1 OUTLINE OF THE METHOD

The viscosity of resins is measured by determining the torque of a spindle rotating at constant speed in the liquid sample, which is adjusted to $25 \pm 0.5^\circ\text{C}$.

A-2 APPARATUS

A-2.1 A water bath maintained at a temperature of $25.0 \pm 0.5^\circ\text{C}$.

A-2.2 600 ml beaker, at least 75 mm (3'') in diameter.

A-2.3 Thermometer graduated in 0.5°C subdivisions at the temperature required.

A-2.4 Brookfield Synchro-electric Viscometer

A-2.4.1 Various models of viscometers are available from Brookfield Engineering Laboratories. Models which are capable of reading accurately viscosity between 10 to 50 000 cps have to be used.

A-2.4.2 Calibration

The calibration of the instrument should be checked periodically by measuring the viscosity of Brookfield Engineering Laboratories viscosity standard fluids.

A-2.4.3 Choice of Spindle and Rotational Speed

The choice of spindle and rotational speed for specific sample should be chosen so that reading will be in the centre of the scale. For thixotropic systems, the variance in the choice of spindle and rotational speed will cause variation in the results. Hence, while reporting viscosity, particularly for thixotropic fluids the spindle number and rotational speed should be mentioned.

A-3 PROCEDURE

A-3.1 Attach the viscometer with an adjustable clamp to a ring stand. Adjust the legs at the base of the ring stand, till the viscometer is properly

levelled. Attach the spindle that applies torque to the range expected for the sample.

A-3.2 Place sufficient sample in a 600 ml beaker to cover the immersion mark on the spindle. Immerse the beaker in the bath to the sample level. Cover the beaker with watch glass to minimize monomer loss. Stir occasionally, without trapping air bubbles to attain uniform temperature throughout. After the desired temperature is reached throughout the sample wait for 10 minutes to get the resin completely stabilized. Immerse the viscometer spindle and guard into the sample to the immersion line marked on the spindle taking care to avoid trapping air during immersion.

Press down the clutch lever and start the motor. Release the clutch lever and allow the dial to rotate until the pointer stabilizes at a fixed position. Depress the clutch lever, stop the motor and read the scale. If the pointer is not in view, switch on and off the motor keeping the clutch lever depressed until the pointer reaches the window.

A-3.3 Repeat the procedure until 3 readings on the scale agree within one percent.

A-4 CALCULATIONS

A-4.1 Multiply readings on the scale by the factors given by the manufacturers to obtain viscosity in centipoise, for corresponding rotational speed and spindle number.

A-4.2 At higher speeds air resistance on the pointer has a certain effect. Compensation shall be made for air resistance as suggested by manufacturer.

A-5 REPORT

A-5.1 The report shall include the following:

- a) Average value of 3 readings in centipoise,
- b) Spindle No. and rotational speed used,
- c) Model of viscometer, and
- d) Temperature of test.

ANNEX B

[Table 1, Sl No. (ii)]

DETERMINATION OF ACID VALUE

B-1 REAGENTS

B-1.1 Alcoholic Potassium Hydroxide—0.1 N.

B-1.2 Indicator

Thymol blue or phenolphthalein indicator solution.

B-1.3 Solvent

Methyl ethyl ketone (MEK), acetone, chloroform or a mixture of them.

B-2 PROCEDURE

B-2.1 Weigh approximately 1 g of resin in a dry 150-ml conical flask and dissolve in 25 ml of solvent. Add two or three drops of indicator and titrate the solution with alcoholic potassium hydroxide.

B-2.2 Carry out a blank determination by titrating 25 ml solvent with the alcoholic potassium hydroxide.

B-3 CALCULATION

B-3.1 Calculate the acid value of the resin as follows:

$$\text{Acid value} = \frac{V_1 - V_2}{M} \times 5.6 \times f$$

where

V_1 = volume in ml of alcoholic potassium hydroxide solution required by the sample,

V_2 = volume in ml of alcoholic potassium hydroxide solution required for the blank,

f = factor for 0.1 N alcoholic potassium hydroxide solution, and

M = mass in g of sample.

B-3.2 Record the mean of the two determinations.

ANNEX C

[Table 1, Sl No. (iii)]

DETERMINATION OF VOLATILE CONTENTS

C-1 APPARATUS

A flat-bottomed dish of nickel, stainless steel or tinplate 7 cm in diameter and 2 to 3 cm deep.

C-2 PROCEDURE

Dry the clean dish for at least 30 minutes in an oven at $100 \pm 1^\circ\text{C}$, cool in a desiccator and weigh to the nearest mg (M_1). Weigh 2.0 ± 0.2 g of the sample by difference from a weighing bottle or weighing pipette into the dish taking care to minimize loss by evaporation during transfer. Record the mass of the sample to the nearest mg (M_2). Spread the contents of the dish as evenly as possible over the bottom of the dish by tilting and if necessary, gently warm the dish to facilitate this. Place the dish and the contents on a level perforated sheet metal shelf in a ventilated oven provided with a fan and maintained

at $150 \pm 2^\circ\text{C}$ for 60 ± 2 minutes. Remove the dish from the oven, allow to cool in desiccator and weigh (M_3). The volatile content of the material under test shall be the mean of the two determinations.

C-3 CALCULATION

Calculate the volatile contents as follows:

$$\text{Volatiles, percent by mass} = \frac{M_2 - M_3}{M_2 - M_1} \times 100$$

where

M_2 = mass in g of the sample + dish before heating,

M_3 = mass in g of the residue + dish after heating, and

M_1 = mass in g of the dish.

ANNEX D

[Table 1, Sl No. (iv)]

DETERMINATION OF GELTIME AT 25°C

D-1 OUTLINE OF THE METHOD

The material, catalyst and accelerator are mixed in an aluminium or stainless steel container and a stirrer is started simultaneously. When the motor used to run the stirrer cuts out, the time is read on the counter.

D-2 APPARATUS

D-2.1 Water-bath maintained at a temperature of $25.0 \pm 0.5^\circ\text{C}$.

D-2.2 Cylindrical aluminium or stainless steel container 45.0 ± 3.0 mm in diameter, not less than 75 mm deep and 0.4 ± 0.1 mm thick.

D-2.3 A thin stainless steel rod about 100 mm long concentric and normal to a circular stainless steel disc at one end 1.5 mm thick with an area between 3.832 to 3.920 cm^2 is connected through a simple mechanical device to an electric motor, so that the disc can be moved up and down in a vertical plane. The total mass of the rod and disc is 16.20 ± 0.02 g. When the motor is switched on, the rod and the disc fall 12.7 mm in $1/2$ minute and then rise again completing the reciprocation in 1 minute. An automatic device counts the number of reciprocations.

A mechanical trip is arranged to cut out the motor at a resistance of 16.4 ± 0.4 g to the movement of the disc.

D-3 PROCEDURE

Clamp the apparatus described in D-2.3 in such a way that the rod and disc can move vertically in the centre of the empty aluminium cylinder, which is immersed in a water-bath to a depth of at least 60 mm. Weigh 100 g of the material to be tested into the aluminium container. Add the amount of accelerator specified by resin manufacturer and thoroughly stir till accelerator is completely dispersed. Avoid the entrapment of air during stirring as far as possible. Now add, the amount of catalyst specified by the resin manufacturer and thoroughly mix it. The moment catalyst is added to the resin, switch on the motor so that the time taken to mix the resin is recorded as part of the gel time of the resin. Adjust the apparatus so that there is complete reciprocation, and in its lowest position the disc is between 12 to 25 mm from the bottom of the container. The motor shall cut out at the first sign of gelation of the resin and the counter gives the geltime of the resin in minutes at 25°C .

ANNEX E

[Table 2, Sl No. (i)]

DETERMINATION OF COLOUR

I HAZEN COLOUR

E-1 OUTLINE OF THE METHOD

The colour of the prepared test solution is compared against known standard colour solution of platinum salts.

E-2 REAGENTS

E-2.1 Chloroplatinic Acid

Dissolve 1.0 g of platinum in a small quantity of aqua regia by heating on a water-bath. When the material has dissolved, evaporate the solution to dryness. Add 4 ml of hydrochloric acid (relative density 1.18) and again evaporate to dryness. Repeat this operation twice. Alternatively take 2.490 g of potassium chloroplatinate crystals (K_2PtCl_6) which corresponds to 1.0 g of platinum.

E-2.2 Cobaltous Chloride—crystals ($\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$).

E-2.3 Hydrochloric Acid—relative density 1.18.

E-2.4 Colour Standard Solutions

Dissolve 2.0 g of cobaltous chloride and either the chloroplatinic acid or 2.490 g of potassium chloroplatinate in 200 ml of the hydrochloric acid, warming if necessary to obtain a clear solution, and, after cooling, pour into a 2-litre measuring flask, and dilute to the mark with water. This solution has a colour of 500 Hazen units. From this solution prepare a series of permanent colour standards having a range of 50 to 300 Hazen units. For this purpose, for each 10 units, pipette 5.0 ml of the platinum solution into a 250-ml measuring flask and dilute to 250 ml. Pour each standard into a clear colourless stoppered bottle. Use the same type of bottle for each standard. Check these standards at intervals against freshly prepared standards.

E-3 PROCEDURE

E-3.1 Siphon about 250 ml of the resin into a dry bottle of the same type as that used for the storage of the colour standards. Select the standard which most nearly matches the sample. Pour the sample into one of a pair of dry matched 50-ml Nessler cylinder until its depth reaches the graduation mark. Pour the colour standard selected into the other cylinder until the levels are equal. Compare the colour of the sample with that of the standard by looking down the tubes against a white back-

ground, taking care to avoid side illumination. Repeat with other colour standards if necessary. Record the value of colour to the nearest 10 units for colours up to 100 units, to the nearest 25 units for colours between 100 and 250 units, and to the nearest 50 units for deeper colours.

E-3.2 If the colour of the sample is of the order of 300 units or more suitable, dilute with a colourless solvent, before attempting to make a comparison.

II GARDNER COLOUR SCALE

E-4 GENERAL

This method covers the measurement of the colour of transparent liquids by means of comparison with arbitrary numbered glass standards.

E-5 APPARATUS

E-5.1 Glass Standards, 18, numbered separately, and having the colour characteristics given in Table 5. A suitable procedure for their calibration is given in E-9. The colour shall be produced by the glass components only.

E-5.2 Glass Tubes, clear, 10.65 mm in inside diameter and about 114 mm in outside length.

E-5.3 Suitable apparatus for comparing sample and standard. The apparatus may be of any design but shall have the following characteristics.

E-5.3.1 Illumination

CIE Illuminant C or its equivalent.

NOTE — Standard Illuminant C intended to represent daylight. Originally, defined as source C, prepared by using Source A (incandescent lamp light, colour temperature 2856 K) with a blue liquid filter. Except in the UV-region, the spectral power distribution agrees well with that of typical daylight. The correlated colour temperature is 6700 K. (CIE Standards made available from U.S. National Committee, CIE, National Institute of Standards and Technology, Gaithersburg MD 20899).

E-5.3.2 Surrounding Field

The field should not differ significantly in brightness from the samples and standards and should be essentially achromatic.

E-5.3.3 Field of View

The specimen and one or more standards should subtend a visual angle of about 2 degree and be in the field of view simultaneously.

E-5.3.4 Separation of Standard and Specimen

There should be a perceptible separation between specimen and standard, but this should be as small as is mechanically possible.

E-6 PROCEDURE

E-6.1 Fill a glass tube with the material under test, if the material is perceptibly cloudy, first filter it.

E-6.2 Compare with glass standards, determining which standard most closely matches the specimen in brightness and saturation. Ignore hue differences.

E-7 REPORT

Report the colour as the number of the standard most closely matching the specimen. If more precise measurements are needed, report as either matching a standard or lighter or darker. Thus, between colours 5 and 6, the steps will be 5; 5+; 6-; and 6.

E-8 PRECISION

E-8.1 Repeatability

Two results obtained by a single operator should be considered suspect if they differ by more than two-thirds of a colour number.

E-8.2 Reproducibility

Two results, each of the mean of duplicate measurements, made by operators in different laboratories should be considered suspect if they differ by more than four-thirds of a colour number.

NOTE — If desired, liquid standards matching the colours given in Table 5, in glass tubes similar to the samples tubes may be used. These may be filled with potassium chloroplatinate for the light colours and solutions of ferric chloride and cobalt chloride in hydrochloric acid for the darker colours.

Table 5 Colour Specifications of Reference Standards
(Clause E-5.1)

Gardner Colour Standard Number	Chromaticity Coordinates ¹⁾		Luminous Transmittance Y (%)	Transmittance Tolerance (±)
	x	y		
(1)	(2)	(3)	(4)	(5)
1.....	0.317 7...	0.330 3...	80	7
2.....	0.323 3...	0.335 2....	79	7
3.....	0.332 9...	0.345 2....	76	6
4.....	0.343 7...	0.364 4....	75	5
5.....	0.355 8...	0.384 0....	74	4
6.....	0.376 7...	0.406 1.....	71	4
7.....	0.404 4....	0.435 2....	67	4
8.....	0.420 7....	0.449 8....	64	4
9.....	0.434 3....	0.464 0....	61	4
10.....	0.450 3....	0.476 0....	57	4
11.....	0.484 2....	0.481 8....	45	4
12.....	0.507 7....	0.463 8....	36	5
13.....	0.539 2....	0.445 8....	30	6
14.....	0.564 6....	0.427 0....	22	6
15.....	0.585 7....	0.408 9....	16	2
16.....	0.604 7....	0.392 1....	11	1
17.....	0.629 0....	0.370 1....	6	1
18.....	0.647 7....	0.352 1....	4	1

¹⁾ A duplicate standard shall have chromaticity coordinates that differ from the reference standard by no more than one-third of the difference in x or y between adjacent reference standards. In any one set, no two standards shall be closer together than two-thirds of the difference in x or y between corresponding reference standards.

E-9 CALIBRATION OF GLASS REFERENCE STANDARDS

E-9.1 Select a dual beam spectrophotometer with a sufficiently small light beam at the sample position so that all rays will pass through the standards to be calibrated. Alternatively equip the spectrophotometer with a condensing lens to accomplish this purpose.

E-9.2 Place the standards in turn in the sample position of the spectrophotometer. If the comparator is provided with a separate green filter in

front of the light source, place this filter in the reference beam of the dual beam spectrophotometer during calibration of each standard.

E-9.3 Obtain spectral transmittance data for each glass reference standard.

E-9.4 From the spectral transmittance data for each reference standard calculate the CIE tristimulus values, X, Y, Z and the chromaticity coordinates, x, y for CIE Illuminant C (see E-5.3.1).

ANNEX F

[Table 2, Sl No. (ii)]

DETERMINATION OF RELATIVE DENSITY

F-1 OUTLINE OF THE METHOD

F-1.1 The relative density is determined with a relative density bottle or pycnometer.

F-1.2 Temperature Corrections

F-1.2.1 In certain cases, it may be inconvenient to determine the relative density at the specified temperature, in which case, it may be determined at a convenient temperature and the results corrected as given in F-1.2.2 and F-1.2.3.

F-1.2.2 If the temperature of testing is higher than the specified temperature, add 0.000 65 to the value

of the relative density obtained for each degree centigrade of the difference between the two temperatures.

F-1.2.3 If the temperature of testing is lower than the specified temperature, subtract 0.000 65 for each degree centigrade of the difference between the two temperatures.

F-2 APPARATUS

F-2.1 Relative Density Bottle or Pycnometer—with well fittings ground-glass joints.

To calibrate, dry the bottle or pycnometer thoroughly, weigh and then fill with freshly boiled and cooled water at about 25°C after removing the cap of the side arm. Fill to overflowing by holding the bottle or pycnometer on its side in such a manner as to prevent the entrapment of air bubbles. Insert the stopper and immerse in a water-bath at the desired test temperature $\pm 0.5^\circ\text{C}$. Keep the entire bulb completely covered with water and hold at that temperature for 30 minutes. Carefully remove any water which has exuded from the capillary opening. Remove from the bath, wipe completely dry, replace the cap, cool to room temperature and weigh. Calculate the weight of water. This is a constant for the bottle or pycnometer, but should be checked periodically.

F-2.2 Water-Bath, maintained at $27 \pm 0.5^\circ\text{C}$.

F-2.3 Thermometer—Any convenient thermometer of a suitable range with 0.1°C or 0.2°C subdivisions.

F-3 PROCEDURE

Fill the bottle with the material previously cooled to about 25°C , holding the bottle on its side in such

a manner as to prevent the entrapment of air bubbles after removing the cap of the side arm. Insert the stopper, immerse in the water bath at $27.0 \pm 0.5^\circ\text{C}$ and hold for 30 minutes. Carefully wipe off any material which has come through the capillary opening. Remove the bottle from the bath, clean and dry it thoroughly. Replace the cap of the side arm, cool to room temperature and weigh.

F-4 CALCULATION

$$\text{F-4.1 Relative density at } 27^\circ\text{C}/27^\circ\text{C} = \frac{A - B}{C - B}$$

where

- A = mass in g of the relative density bottle with polyester resin at 27°C ,
- B = mass in g of the relative density bottle, and
- C = mass in g of the relative density bottle with water at 27°C .

F-4.1.1 Temperature Corrections

If the relative density is determined at any other temperature, then temperature corrections as given under F-1.2 shall be applied.

ANNEX G

[Table 2, Sl No. (iii)]

METHOD OF DETERMINING REFRACTIVE INDEX

G-1 PRINCIPLE

The refractive index is obtained by the measurement of the deviation using a refractometer caused by the liquid to a light beam passing through it.

G-2 APPARATUS

G-2.1 Abbe refractometer with scale graduated directly in terms of refractive index of the D-line of sodium at a temperature of 23°C .

G-2.2 Water supply, the temperature of which may be varied.

G-2.3 A source of sodium light.

G-3 PROCEDURE

Place the refractometer in front of the source of sodium light, insert the thermometer and adjust the circulation of water so as to bring the prisms to the desired temperature (usually 25°C). Clean the prisms with alcohol and wipe dry. Spread a drop of the liquid to be tested upon the lower prism and clamp the prisms together. Adjust the mirror so that the light enters the microscope. Focus the eyepiece on the cross-hairs and the reading lens of

the scale. Upon moving the prism arm a position can now be found where the lower part of the field is dark and upper part light. In general, the borderline will be coloured. Correct by turning the milled head on the right of the telescope so that a sharp black and white edge is obtained. Move the prism arm until this black edge just crosses the intersection of the cross-hairs. Read the refractive index from the scale, estimating the fourth decimal place.

The accuracy of the instrument may be checked by a small test plate of known refractive index, which is supplied with the refractometer. Attach this test plate to the upper prism with liquid of high refractive index (usually monobromonaphthalene). Errors may be corrected by means of small adjusting screw.

CAUTION — Care must be taken in the use of monobromonaphthalene as it may be absorbed through contact skin. It will cause irritation to the skin and eyes on prolonged contact and nausea and vomiting if swallowed in significant quantities.

G-4 Report the refractive index determined in G-3.

ANNEX H

[Table 2, Sl No. (iv)]

DETERMINATION OF GEL TIME AT 82°C

H-1 APPARATUS

The apparatus shall be assembled as shown in Fig. 1:

- 150-ml round-bottom flask (A) with a ground-glass back,
- 300-mm Leibig condenser with ground-glass joint,
- Adaptor,
- 152 mm × 25.4 mm test-tube (B) of heat-resistant glass containing 6 to 7 g of liquid paraffin,
- 152 mm × 19 mm test-tube (C), and
- 6.0 mm dia glass stirring rod.

NOTE — A suitable constant temperature bath may also be used instead of apparatus as described at (a), (b), (c) and (d).

H-2 PROCEDURE

H-2.1 Keep 50 ml of isopropyl alcohol (see IS 2631 : 1976) boiling in the flask A, so that the temperature of the liquid paraffin in the test-tube B is maintained at 81 to 82.5°C. Catalyse 100 g of the polyester resin to be tested in the proportions specified by the resin manufacturer to an accuracy of 2.5 percent of those proportions, in accordance with the manufacturer's instructions. Pour a layer of the resin approximately 25 mm deep into test tube C which shall be immediately assembled in the apparatus as shown. Start a stop-clock at the moment test-tube C is immersed in the liquid paraffin. Place the stirring rod in the resin, and stir the resin

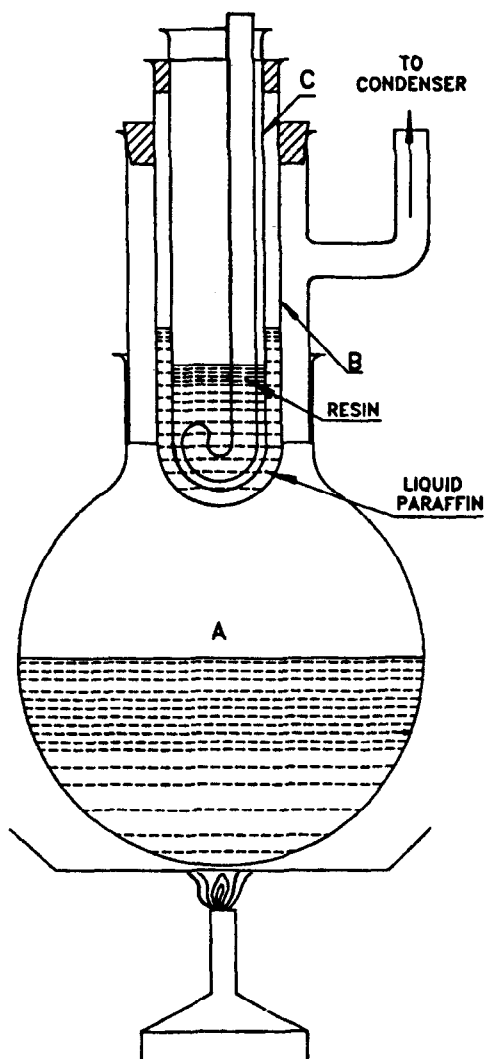


FIG. 1 GEL TIME APPARATUS

by making one complete revolution of the rod around the approximate circumference of the resin in the test-tube *C* once every 15 seconds, until gelation occurs.

H-2.2 The gel time of the resin, measured from the time of immersion in the hot liquid paraffin, shall be taken to be that at the end of which the resin just ceases to be fluid.

ANNEX J

[Table 3, Sl No. (i)]

DETERMINATION OF BARCOL HARDNESS

J-1 OUTLINE OF THE METHOD

With the help of Barcol Impressor [Model No. GY2J 934.1, made available from M/s Barber Colman Co, Rockford, Illinois (USA) or any equivalent model from any source] the hardness, that is material resistance to indentation, is measured. Both reinforced and unreinforced specimens hardness can be measured.

J-2 APPARATUS

J-2.1 Indentor

The indentor shall consist of hardened steel truncated cone, having an angle of 26°C with flat tip of

0.157 mm diameter.

J-2.2 Indicating Device

The indicating device shall have 100 divisions each representing a depth of 0.007 6 mm penetration — the higher the reading the harder the material.

J-2.3 A typical diagram of Barcol Compressor is shown in Fig. 2.

J-3 TEST SPECIMENS

J-3.1 Resins should be cast as suggested in Annex P. Test pieces shall be at least 1.5 mm thick and large enough in area to ensure a minimum distance of 3 mm in any direction from the point of measurement to the edge of the specimen. The

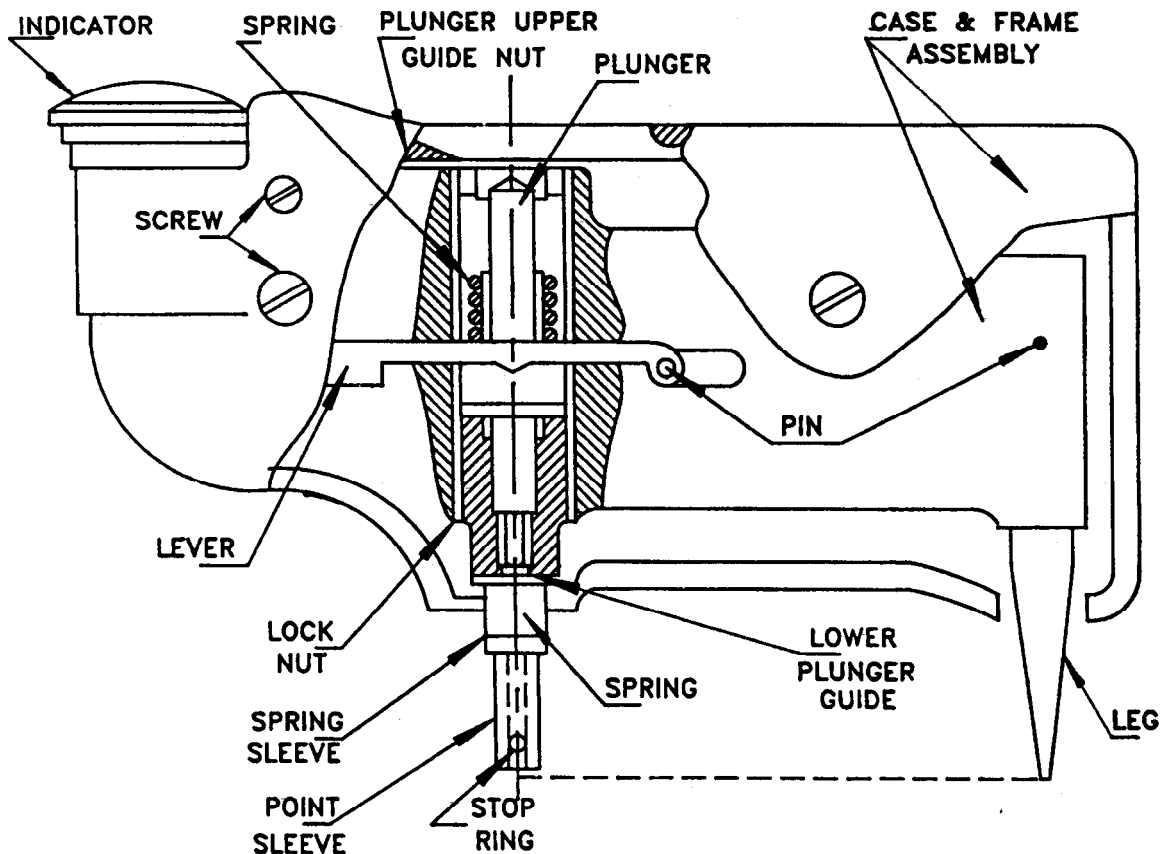


FIG. 2 BARCOL COMPRESSOR

specimens should be cured as suggested by the supplier of the resin.

J-3.2 Test specimens should be conditioned at $27 \pm 2^\circ\text{C}$ and $65 \pm 5\%$ relative humidity for not less than 25 h prior to testing.

J-3.3 Tests should be conducted in standard laboratory atmosphere of $27 \pm 2^\circ\text{C}$ and $65 \pm 5\%$ relative humidity.

J-3.4 Calibration of Barcol Impressor

As suggested by the manufacturer of Barcol Impressor.

J-4 PROCEDURE

J-4.1 Place the impressor and the material to be tested on a flat and hard surface. Set the point sleeve on the surface to be tested. Set the legs on the same surface or on solid material of the same thickness. Grasp the instrument firmly between the legs and the point sleeve. Apply quickly, by hand, sufficient pressure downwards and note the highest dial reading. Take care to avoid sliding while the indenter is in contact with the surface being tested.

J-4.2 Take a minimum of 8 readings, taking care that impressions are not made within 3 mm edge of specimen or earlier impressions.

J-4.3 Report the average of hardness values.

ANNEX K

[Table 3, Sl No. (iii)]

WATER ABSORPTION OF CAST RESIN

K-1 OUTLINE OF THE METHOD

The test specimen is immersed in water for specified period. After removing surface water the amount of water absorbed is formed by difference in weight.

K-2 PREPARATION OF TEST SPECIMEN

Three specimens of 50 ± 1 mm square shall be machined from cast sheet prepared in accordance with Annex P. The thickness of cast sheet shall be 3.0 ± 0.15 mm.

K-3 PROCEDURE

K-3.1 Dry the specimen in an oven for 24 ± 1 h at $50 \pm 2^\circ\text{C}$, cool in a desiccator and weigh to the

nearest mg (M_1). Place the specimen in a container of distilled water maintained at a temperature of $27 \pm 2^\circ\text{C}$. Precaution shall be taken to prevent specimens from making contact over any substantial area with one another or with the container. After 7 days take them from the water and remove the surface water with a clean dry cloth or with filter paper. Weigh the specimens within one minute of taking them from the water (M_2).

K-3.2 The water absorption of the test specimen shall be $(M_2 - M_1)$ and shall be expressed in mg. Repeat the water absorption of the material under test as the arithmetic mean of the various tests obtained on the test specimens.

ANNEX L

[Table 2, Sl No. (v)]

DETERMINATION OF HYDROXYL VALUE

L-1 GENERAL

This method determines the difference between the hydroxyl value and the acid value; it is therefore necessary to determine the acid value separately, in order to calculate the hydroxyl value.

NOTE — The hydroxyl value of saturated polyester resins (for example, polyester resin used for the manufacture of polyurethanes and polymeric plasticizers) and of certain types of alkyd resins may also be determined by this method.

L-2 DEFINITION

L-2.1 Hydroxyl Value

The number of milligrams of potassium hydroxide necessary to neutralize the acetic acid which will

combine by acetylation with 1 g of the unsaturated polyester resin.

L-3 PRINCIPLE

L-3.1 Acetylation of the hydroxyl groups by acetic anhydride is carried out on an ethyl acetate solution of the resin in the presence of toluene-4-sulphonic acid catalyst. The excess acetic anhydride is hydrolyzed by a pyridine/water mixture and the resultant acetic acid titrated with standard volumetric methanolic potassium hydroxide solution.

In this titration, the free acid groups which exist in the resin are also neutralized by the potassium

hydroxide. The hydroxyl value is finally calculated by taking into account the acid value determined separately.

L-4 REAGENTS

L-4.1 Acetic Anhydride Acetylating Solution—approximately 1 M, in ethyl acetate.

Dissolve 1.4 g of pure, dry toluene-4-sulphonic acid in 111 ml of anhydrous ethyl acetate. When completely dissolved, slowly add, while mixing, 12 ml of freshly distilled acetic anhydride and store in a dry atmosphere.

L-4.2 Ethyl Acetate—anhydrous.

L-4.3 Pyridine/Water Mixture—3 + 2 (V/V).

Mix 3 volumes of pyridine, analytical reagent quality, with 2 volumes of water.

L-4.4 Butanol-1/Toluene Mixture—2 + 1 (V/V).

L-4.5 Mixed Indicator Solution

Mix 3 volumes of a 0.1% ethanolic solution of thymol blue with 1 volume of a 0.1% ethanolic solution of cresol red.

L-4.6 Potassium Hydroxide

0.5 N standard volumetric solution in methanol.

L-5 APPARATUS

L-5.1 Conical Flask—capacity 250 ml, with a ground glass stopper.

L-5.2 Magnetic Stirrer—with a magnetic bar covered with a corrosion-resistant material (for example PTFE).

L-5.3 Burette—capacity 50 ml, graduated in 0.05 ml.

L-5.4 Water Bath—controlled at a temperature of $50 \pm 1^\circ\text{C}$.

L-5.5 Pipette—capacity 10 ml (for the acetylating solution).

L-5.6 If necessary : Apparatus for potentiometric titration.

L-6 PROCEDURE

Weigh, to the nearest 1 mg, in the 250 ml conical flask (L-5.1), a test portion of the resin containing approximately 5 milli-equivalents of OH (the mass of grams of the sample = $280/\text{hydroxyl value}$).

NOTE — If the approximate hydroxyl value is not known, preliminary tests should be made.

Add exactly 10 ml of the acetylating solution (L-4.1) and the magnetic bar (see L-5.2).

Stopper the conical flask after moistening the stopper with ethyl acetate (L-4.2) and dissolve the test portion, using the magnetic stirrer (L-5.2).

NOTE — Should the sample not dissolve completely on warming, 5 or 10 ml of the acetylating solution may be added.

Place the conical flask in the water bath at $50 \pm 1^\circ\text{C}$, taking care to immerse it only about 10 mm, and leave it for 45 min.

NOTE — This time may be reduced, for example to 30 minutes or less, provided it can be shown that equivalent results are obtained.

Remove the conical flask from the bath, cool, place on the magnetic stirrer and add 2 ml of distilled water. When the solution has been thoroughly mixed, add 10 ml of the pyridine/water mixture (L-4.3) and stir for 5 min.

Rinse the stopper and inner surface of the conical flask with 60 ml of the butanol-1/toluene mixture (L-4.4) and add 5 drops of the mixed indicator solution (L-4.5).

Continue stirring and titrate with the methanolic potassium hydroxide solution (L-4.6). When the colour change is observed, add a further 1 to 2 drops of the mixed indicator. The solution changes from yellow to clear: note the volume V_1 , in millilitres, of potassium hydroxide solution used. Add a further drop of the potassium hydroxide solution; the indicator colour should turn to blue. If it does not, note the burette reading and add a further drop of the mixed indicator solution; continue in this way until the blue colour is obtained.

The value V_1 to be used for the calculation is the one noted before adding the drop which produced the blue colour.

Carry out a blank test under the same conditions; but without the test portion, and note the volume V_2 , in millilitres, of potassium hydroxide solution used.

At least two tests should be carried out. The results of the two tests should not differ by more than 2 units of the hydroxyl value. If this is not the case, further tests should be carried out until the results of two consecutive tests fulfill the requirement.

NOTE — It is possible, as an alternative, especially when dealing with densely coloured products, to replace the titration in the presence of an indicator by a potentiometric titration. Use a calomel reference electrode with a bridge containing a saturated solution of potassium chloride in methanol and a glass electrode connected to a pH meter or to millivoltmeter.

L-7 EXPRESSION OF RESULTS

From each of the two results obtained, calculate the hydroxyl value by the formula

$$\frac{(V_2 - V_1) \times N \times 56.1}{m} + A_v$$

where

- A_v is the acid value¹⁾;
 V_1 is the volume, in millilitres, of standard of volumetric potassium hydroxide solution used in the determination;
 V_2 is the volume, in millilitres, of standard volumetric potassium hydroxide solution used in the blank test;

- N is the normality of the standard volumetric potassium hydroxide solution (usually 0.5 N); and
 m is the mass, in grams, of the test portion.

Calculate the average of the two values obtained to the nearest unit.

NOTE — The value of $(V_2 - V_1)$ can be positive or negative.

ANNEX M

[Table 2, Sl No. (vi)]

METHOD FOR DETERMINING THE CUMULATIVE LOSS OF STYRENE MONOMER

M-1 PRINCIPLE

A single layer of chopped strand mat (CSM) is impregnated with the catalysed resin and supported on an accurate direct reading top pan balance in a temperature controlled draught free room. The styrene loss is assessed from the rate of mass loss per unit area.

M-2 APPARATUS AND MATERIALS

M-2.1 Top panel direct reading balance with an accuracy of 0.01 g.

M-2.2 Polyethylene terephthalate (PET) release film.

M-2.3 Glass fibre chopped strand mat (CSM), emulsion bound, with a weight per unit area of 450 g/m².

M-2.4 Draught free room, having a minimum capacity of 50 m³, controlled at 27 ± 2°C.

M-2.5 Optional shielding as shown in Fig. 3.

M-2.6 Glass Plate, 3 mm to 4 mm thick and of area 300 mm × 300 mm.

M-2.7 Glass Beaker, capacity 500 ml.

M-2.8 Spatula

M-3 PROCEDURE

M-3.1 Set the apparatus in the draught free room.

NOTE — As it is possible that draughts will be encouraged, it is recommended that shielding should be used. The shielding should be positioned such that there is a minimum clearance between the platten and the shield of 100 mm.

M-3.2 Cut a 250 mm × 250 mm section of CSM and determine its mass. Cut a 300 mm square sample of release film and mark area of 250 mm × 250 mm on the back of the film with a marker pen. Adjust the resin temperature to 27°C.

M-3.3 Add the amount of catalyst as recommended by the manufacturer to the resin in a glass beaker, and if required, accelerator to give a gel time of 15 min to 20 min determined in accordance with Annex D and mix well. Place the release film on the glass plate. Pour a quantity of resin from the glass beaker onto the release film equal to two and half times the mass of the CSM and spread it evenly over the marked area.

M-3.4 Place the CSM sample into the resin and quickly ensure complete impregnation of the mat by gently pressing with the edge of a spatula. Within 1 minute place the impregnated mat and the glass plate on the balance and note the reading.

M-3.5 Record the mass at 2 minutes, 5 minutes, 10 minutes and thereafter at 10 minutes intervals for 1 h and at 30 min intervals to constant mass. Report the procedure twice more.

M-4 EXPRESSION OF RESULTS

From the results plot a graph of mean cumulative loss in mass against time and read off the cumulative loss of styrene (in g/m²) at 60 min and other times, if required.

¹⁾ It should be noted however that the results obtained correspond to only half of the free anhydrides. In the case of resins based on maleic anhydride/hexachloro-endomethylene-tetrahydrophthalic acid and maleic anhydride/tetrahydrophthalic acid, the error is very small, being slightly greater in the case of resins of maleic anhydride/orthophthalic acid type.

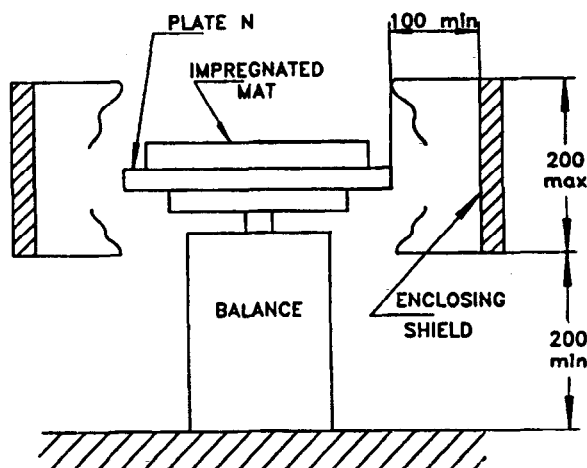


FIG. 3 OPTIONAL SHIELDING

ANNEX N

[Table 4, Sl No. (i)]

METHODS OF TEST FOR FLAMMABILITY OF CAST RESIN

N-1 OUTLINE OF THE METHOD

The cast sheet is subjected to a flame for specified time from Bunsen burner and its flammability evaluated.

N-2 PREPARATION OF SPECIMEN

From the cast sheet prepared in accordance with Annex P, four specimens shall be cut not less than 150 mm long, 12.0 ± 0.5 mm wide and 3.0 ± 0.15 mm thick and a line shall be drawn, across the specimen at 75 mm from the end to be ignited, at right angles to the longitudinal axis.

N-3 PROCEDURE

Test the specimen in a draught-free atmosphere. Clamp it in a rigid support at one end so that its longitudinal axis is horizontal and its transverse axis is at 45°C to the horizontal, and the line on the specimen is clearly visible. Clamp a piece of clean wire gauze (7 mesh per linear cm) 125 mm square in a horizontal position 6 mm below the specimen with 6 mm of the unsupported end of the specimen projecting beyond the edge of the gauze. Place a burner centrally under the free end of the specimen

so that the top of the burner is 50 mm vertically below the longitudinal axis of the specimen. The temperature of the flame shall be such that a piece of 0.70 mm diameter copper wire held 5 cm above the top of the burner melts within 6 seconds. Remove the flame after 30 seconds and record the time taken until the specimen ceases to flame or glow. In the event of the specimen extinguishing within 10 seconds of the removal of the flame, apply again the flame for a further 30 seconds.

N-4 ASSESSMENT

N-4.1 Very Low Flammability

At least three or four specimens shall cease to flame or glow within 10 seconds of the removal of the flame and again within 10 seconds after the second removal of flame.

N-4.2 Low Flammability

At least three of the four specimens shall cease to flame or glow before the flame reaches the 75 mm mark.

ANNEX P

[Clause 4.5]

CHEMICAL RESISTANCE TEST

P-1 PREPARATION OF UNFILLED RESIN CASTINGS

P-1.1 Cast the resin to be tested in the form of an unfilled casting 3.00 ± 0.15 mm (for flammability

and water absorption) or 0.50 ± 0.25 mm (for deflection temperature under load) using the catalyst or catalyst/accelerator system and curing schedule specified by the resin manufacturer.

P-1.2 A suitable mould for casting is made from two pieces of plate and a suitable gasket 25 mm wide. Clamp the pieces of plate-glass and gasket after covering with a suitable release film in a vertical position with the gasket in between to form an open ended cell.

P-1.3 A suitable gasket can be made out of polyvinyl chloride or polyethylene sheet.

P-2 TEST PIECE

Prepare the casting as detailed in P-1.

P-3 PROCEDURE

P-3.1 Cut three specimens $50 \times 50 \pm 1$ mm from the casting. Determine the length, width, thickness and weight of each piece accurately and immerse

the samples in the appropriate liquid, care being taken to ensure that all the surfaces of the samples are in contact with the liquid. Carry out the test at 95 to 100°C or at the boiling point of the liquid, whichever is the lower, or at any other temperature which may be agreed. Carry out the test for 1, 2, 4, 8 days, and on removal from the test liquid, immerse the specimens in the same liquid at $20 \pm 5^\circ\text{C}$ for 15 minutes. Remove, wipe dry test immediately for the four parameters determined previously. In the case of corrosive media wash the specimens in cold water or other suitable solvent for 15 seconds and subsequently wipe dry before testing.

P-3.2 For each parameter quote the mean of the results on the three specimens, report the test temperature also.

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This Indian Standard has been developed from Doc : No. PCD 12 (1208).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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